Melting Point Determination
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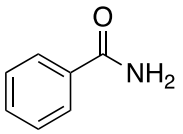
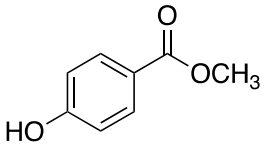
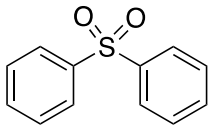
A melting point is an intrinsic property of a pure compound and as such it can be used to not only help identify the compound but also help determine whether or not a given sample of that compound is pure. *[An intrinsic property is a property that is specific to a pure compound regardless of the source of that compound. Intrinsic properties include color, melting point, boiling point, density, refractive index, molecular weight, optical rotation and spectral data (IR, NMR, UV and mass)].* The melting point of a pure compound is a very narrow range (1-3°C) and represents the highest temperature at which that compound can melt. When a compound is impure, its melting point is lower than that of the pure material and the melting point range is usually noticeably larger than that of the pure material. In today's experiment, you will learn to take melting points and use the observed melting points to determine whether a given sample is pure or impure. You will also learn to determine the identity of an unknown, pure compound by taking its melting point and then comparing this value with the melting points obtained after it has been mixed individually with three different known compounds. It should be apparent from the above discussion that if a compound is mixed with another sample of itself, the compound remains pure and the melting point is unaffected. Conversely, if a compound is mixed with a different structure, it becomes impure and the melting point is lowered and the melting point range broadens.



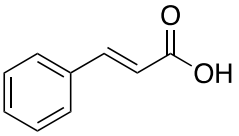
Benzamide



Methyl 4-hydroxybenzoate



Diphenylsulphone



trans Cinnamic acid

Today's experiment will involve some or all of the above compounds, each of which melts somewhere in the 120-135°C range. Determine the melting point of each of the above pure compounds. You will be given samples of three unknown compounds. Determine the melting points of each of these compounds and using these melting points decide whether or not these samples are pure. For the pure unknown compound, determine its identity by taking mixed melting points with each of the known compounds shown above.

To take a melting point, place a small sample (.03 g is plenty) on a clean watch glass and use the closed end of a clean, small test tube to crush the sample into powder form. Pack a sample of this material into the closed end of a capillary tube. The height of this packed material should be between 2-4 mm. *[If too much material is placed in the tube, the melting point range will be erroneously large].* Place the packed capillary tube into the melting point apparatus and determine the melting point. The melting point is a range: the temperature at which the first drop of liquid appears to the temperature at which the entire sample is liquid. The ΔT rate of the apparatus should be about 1-3 °C/minute at the melting point. *[To determine the melting point of an unknown compound, place the sample into the apparatus and ramp the temperature up at a rapid rate (10-20°/minute) to obtain an approximate melting point.*

Allow the apparatus to cool to 10° below this point, insert a new sample and slowly ramp up the temperature to determine the actual melting point. If the approximate melting point is known, allow the instrument to rapidly heat to 10-15° below this temperature then start the melting point]. There are some practical realities about taking melting points. First almost all samples of pure compounds are obtained by recrystallization from a solvent. Some of this solvent is inevitably trapped on or in the crystal lattice. Unless the sample is dried under a vacuum usually at a raised temperature, some of this trapped solvent remains. As the temperature is raised during the melting process, the trapped solvent escapes with the appearance that the compound is sweating. This is not the start of the melting point. Many compounds tend to soften as the melting point is approached. Again, this is not the start of the melting point. Look for actual collapse of the crystal structure with actual liquid forming around the solid. Usually, you will see movement of the solid as the crystal lattice begins to collapse. This is the start of the melting point.

To take a mixed melting point, place about .03 g of your unknown and .01 g of one of the known compounds on a clean watch glass. Using the closed end of a clean test tube thoroughly mix and crush these two materials into a powder. Use this material to obtain a melting point in the normal manner.